OPERATING INSTRUCTIONS
RC102 and RC110 CRYOGENIC WORKSTATIONS
Continuous Flow Cryostats

INTRODUCTION

RC102 and RC110 FLOW CRYOSTATS

The RC102 and RC110 cryogenic system transfers liquid helium or nitrogen from a storage dewar through a highly efficient transfer line to the cryostat sample mount.

The system features:

***Fast cooldown**
***Ultra-stable temperatures at the sample mount**
***Extremely fast response to changes in temperature**
***High performance with either liquid helium or nitrogen**
***No shielding flow losses**
***Disconnect of transfer system from cryogenic WORKSTATION.**

The standard RC102 is a fast cooling optical flow cryostat. The RC110 is similar and features an additional temperature stabilizing phase separator.

The typical experimental arrangement will consist of a RC102 or RC110 system, a liquid helium storage dewar, helium gas cylinder with pressure regulator, and a vacuum pump.

The storage dewar insert leg will be inserted into the liquid cryogen. A small pressure is used to transfer the liquid out of the storage dewar into the transfer line and directly to the sample mount. The flow control valve regulates the flow which is set externally using the valve control knob.

Pressure inside the storage dewar is adjusted using a helium gas cylinder containing helium gas; 'welding grade' purity is suitable. A gas regulator should be used, with a sensitivity of 0-5 psig. The transfer line insert leg will seal to a 0.5 inch quick connect, located at the top of the storage dewar.

The RC 102-110 features quick disconnect for transfer line /WORKSTATION separation - just remove the clamp and withdraw the transfer line.

The phase separator regulator (RC 110 only) will adjust a thermal link built inside of the sample mount; this will allow the system to completely eliminate temperature instability in the 5 to 20 K region which can be caused by two-phase liquid-vapor cycling. Electrical connections to the sample & mount are made through the O-ring sealed ports located on the instrumentation housing. Provisions are provided for future instrumentation requirements.

The evacuation valve for the transfer system is located above the valve control knob. The evacuation valve for the sample region is located on the instrumentation housing. The vacuums are completely independent. Remember the vacuum shroud will not be removable until the sample region vacuum is released. DO NOT release the transfer line vacuum.

Sample access is achieved by removing the quick disconnect clamp. The radiation shield can be removed by rotating counter-clockwise (RC110) or unbolting (RC102). On the RC110 single thread radiation shield interface, A SMALL AMOUNT OF CONDUCTIVE HIGH VACUUM GREASE OR TEFLON LUBRICANT SHOULD BE MAINTAINED ON THREAD.

Four 1/4-20 mounting holes are provided on the bottom of the vacuum shroud for rigid mounting of the WORKSTATION.

(8) #4-40 mounting holes are provided on the sample mount for attaching your sample. Take care to minimize the thermal resistance between sample and mount. Usually direct (dry) mounting of samples onto the cold finger is satisfactory. However, a sheet of indium between sample

and mount is generally an excellent interface. Remember to use stainless steel screws and anchor all wiring to the 1.000" OD vent tube.

A <u>wound</u> <u>heater</u> is installed on the sample mount (or at the end of the transfer system with the UHV option). The heater will be used to raise the temperature during variable temperature operation.

A provision for mounting a temperature sensor is located on the sample mount. During short runs the radiation shield will remain relatively warm, since it is cooled by the enthalpy of the exiting vapor. Therefore, it is important to shield the sensor from the radiation of the warm shield. Wrap aluminum foil over the sensor and anchor the foil to the copper section with vacuum compatible tape or low temperature epoxy.

FOLLOW STANDARD AND PROPER CRYOGENIC PROCEDURES. If you have any questions, we, at CRYO Industries, are only a phone call away and would be happy to answer any questions - (603) 621-9957
fax: (603) 621-9960
email:
cryo@cryoindustries.com.

<u>OPERATION</u>

1. EVACUATE TRANSFER LINE

A recommended practice is to evacuate the transfer line, with a high vacuum type pumping system, prior to EACH use. Since a high vacuum system is not always accessible, your model RC 102-110 contains a charcoal cryopump which allows the system to operate with a mechanical `roughing' pump evacuation.

2. EVACUATE WORKSTATION

Attach the vacuum pump to the WORKSTATION evacuation valve located on the instrumentation housing. Evacuate the sample region. Remember that the sample vacuum and transfer system vacuum are completely separate.

3. CHECK FLOW CONDUCTANCE & PURGE TRANSFER LINE

Open the helium throttle valve by turning counter-clockwise the flow valve operator, several turns. Attach a hose from the helium gas cylinder to the exit end of the transfer line. Flow helium gas through the system. Check for helium exiting at the flow control valve. The exiting gas indicates that an open flow system is present. In addition, the transfer line will be purged with helium gas which should prevent any non desirable freezing from occurring. Remove the gas hose from the transfer line. CLOSE THE THROTTLE VALVE `FINGER' TIGHT, DO NOT OVER TIGHTEN.

4. CLOSE PHASE SEPARATOR REGULATOR (RC 110 only)

Close the phase separator regulator; rotate the regulator knob, located above the instrumentation housing, clockwise [looking down on the system from above] as far as the adjustment will allow. This position of the

regulator is used during cooldown.

5. INSERT TRANSFER LINE LEG INTO STORAGE DEWAR

Insert the transfer line into the neck of the storage dewar. Loosely seal the line to the storage dewar quick connect. SLOWLY lower the leg into the ullage space of the storage dewar. Open the flow control valve 3-5 turns. Continue to SLOWLY lower the transfer line leg. At this point two conditions are possible concerning the storage dewar; follow the procedure corresponding to your status. (I). The storage dewar vent is open to atmosphere. Feel for helium gas to exit the storage dewar caused by the transfer line leg entering the liquid. Temporarily, hold the line at this position. Allow the exiting gas to cool the leg section above the liquid. Check the flow control knob to assure that it has not frozen and still rotates. When the venting settles down, continue to slowly insert the leg. Upon reaching the bottom, pull the leg up approximately 0.5" and tightly seal the leg. Attach the helium gas hose to the storage dewar and pressurize to approximately 1.0 psig. (ii). The storage dewar is pressurized and the leg was inserted through a valve, maintaining storage dewar pressure. SLOWLY lower the transfer line leg into the storage dewar. Check the flow control knob to assure that it has not frozen and still rotates. Continue to slowly insert the leg AND CHECK SYSTEM FOR FLOW. Upon reaching the bottom, pull the leg up approximately 0.5" and tightly seal the leg. Attach the helium gas hose to the storage dewar. A pressure between 1 to 5 psig is normal. Pressure setting is not critical. Adjust the pressure if it is outside of the normal range.

6. CHECK SYSTEM FOR FLOW

The valve control knob should be maintained 3-5 turns open. Feel for helium gas exiting the transfer system, or at the helium vent port of the WORKSTATION if the transfer line is connected to the WORKSTATION. Gas exiting indicates an open flow system is present, i.e., nothing has frozen in the valve or transfer line. Connect the RC110 transfer line to the WORKSTATION. Just insert and clamp. Note: the transfer line can be clamped to the WORKSTATION at any prior point, should this be preferable.

7. COOLDOWN

If equipped, MAKE SURE that the phase separator regulator is rotated clockwise [looking down onto the system from above] as far as the adjustment will allow (Knob closed, i.e, no threads showing.) The sample will begin to cool. Average cooldown time to 4.2 K is 8 min (10 min with UHV model). After sample cooldown, the flow valve knob should be rotated to approximately 1 turn open; fine adjust for minimum liquid helium flow along with sufficient cooling power..

8. CHECK STORAGE DEWAR PRESSURE

Maintain a check on storage dewar pressure,

adjust if necessary. Exact pressure settings are not required. After cooldown, the nominal operating pressure is 1 psig.

9. VARIABLE TEMPERATURE Attach a temperature controller or power supply to the electrical feedthrough wired to the heater and located on the instrumentation housing (or transfer line housing for UHV model). The temperature can be varied from 5 to 300 K by adjusting the power input to the heater and/or the flow rate. The rate of temperature increase will vary with the power supplied by the control heater; DO NOT EXCEED 50 watts. Lower temperatures can be obtained by turning off the electrical current supplied to the heater. If desired, more rapid temperature reduction can be obtained by increasing the flow rate (and/or rotating the phase separator regulator clockwise, if in use). Knob settings may be returned to the previous steady state values as the new desired temperature coordinate is approached.

10. PHASE SEPARATION (RC110 ONLY)

The model RC110 has the ability to remove all instability caused by two phase (vapor - liquid) flow. Basically, heat transfer to the flowing liquid helium in the transfer line vaporizes part of the liquid forming vapor which cannot escape or vent from the transfer line. Thus, a liquid - vapor mix flows to the sample mount. This two-phase flow combined with the extremely low heat capacities which occur below 15 K may result in unstable temperature coordinates.

At temperatures above 10 - 15 K, the heat capacities become sufficiently large and the flow requirements may be reduced so as to deliver single phase gas to the sample mount eliminating the temperature fluctuations.

The RC110 utilizes an internal phase separator which completely eliminates all temperature instability in the difficult to control temperature region between 6 and 15 K where two-phase flow causes problems. This technology results in a system with ultra stable temperature coordinates, quicker response time and improved performance with liquid nitrogen.

To engage the phase separator, rotate the separator regulator knob COUNTER CLOCKWISE [looking down on system from top] ALL THE WAY UP until it will no longer turn and tighten The temperature can be varied by adjusting the power input to the heater. A slight increase in flow rate may be beneficial to compensate for the thermal resistance of the phase separator.
Note: The phase separator will fill with liquid cryogen, if cooled to the boiling point of the liquid. The separator must be cleared when power is initially applied to the heater. <u>Using liquid helium, this is probably not noticeable.</u> When operating with liquid nitrogen and current is initially supplied to the heater, a small increase in temperature will occur; followed by a time delay

of a couple of minutes, after which the temperature will again increase; henceforth, no thermal lag will exist. If temperature stability is satisfactory, the phase separator need not be used and can remain in the closed position used during cooldown.

11. ALTERNATE PROCEDURE, VARIABLE TEMPERATURE [15 TO 300 K]
When operation above 15 K, an alternate procedure for operation may be followed which will conserve liquid helium. Rotate the phase separator to the lowest position. Reduce the liquid helium flow rate using the flow control knob. A small liquid helium flow rate and the control heater will maintain the required temperature setting. The flow rate may be reduced further as the temperature is increased.

12. OPERATION BELOW 4.2 K
Attach a mechanical vacuum pump to the helium vent port. Temperature changes are made by adjusting the helium flow valve control knob or control heater.

13. WORKSTATION ROTATED MORE THAN 90 DEGREES
Operation with the vacuum shroud rotated greater than 90 degrees from the normal vertical position will require an increase in the liquid helium flow rate. A nominal setting of the control knob is 1.5 - 2 turns open. Fine adjust the setting after gaining experience with the system. Set the phase separator to its lowest position. After cooldown, slowly rotate the phase separator counter-=clockwise and observe the terminal temperature. Set the knob at the point where the lowest terminal temperature was observed. Adjust the helium flow control knob for the required refrigeration.

14. LIQUID NITROGEN OPERATION
When using liquid nitrogen instead of helium the operation procedure is identical, except the knob settings may vary. The system is designed to eliminate the problems normally encountered due to the high latent heat of vaporization of liquid nitrogen.

15. SHORT TERM SHUT DOWN
If a new experiment is to begin shortly after completion of a `run', the transfer line insert leg can remain in the storage dewar. The phase separator regulator should be rotated to the lowest position and the helium flow valve closed. If preferred, the RC110 line can be removed from the WORKSTATION. The storage dewar pressure can remain. Restart the system opening the helium flow valve - 3 to 5 turns.

16. AFTER `RUN' SAMPLE ACCESS
If the sample is immediately accessed, frost will form on the radiation shield and sample. Frost will not affect the WORKSTATION except for a potential difficulty in removing the radiation shield. However, condensation should be removed before

re-evacuation the WORKSTATION. A heat gun is a useful tool for removing frost and moisture.

It may be preferable to warm the sample region to near room temperature before opening the WORKSTATION by (I). just waiting for the system to return to room temperature. (ii). `Breaking' the shroud vacuum with dry helium gas or nitrogen gas when sample temperature exceeds 80 K. (iii). Applying power to the control heater. DO NOT EXCEED 50 watts. (iv). Flowing warm gas from the helium gas cylinder into the transfer line inlet port of the WORKSTATION.

(RC110 only) Close the flow control valve. If the Storage dewar has a provision for removing the transfer line insert leg without de-pressurization, remove the line. Otherwise, de-pressurize the storage dewar. During pressure reduction, helium gas will vent from the storage dewar. AVOID CONTACT WITH THE COLD GAS. When the dewar returns to atmospheric pressure, remove the transfer line insert leg.

GENERAL NOTES AND SAFETY PRECAUTIONS

1. It is recommended that the transfer line and sample vacuum shroud be evacuated prior to EACH new use with a diffusion type pumping system. Prior overnight `rough' pumping of the <u>transfer</u> <u>line</u> is beneficial.

2. Do not bend the transfer line to less than a 12 inch radius.

3. Heater input power should not exceed 50 watts. If using a temperature controller check setting of current limiter (if so equipped) so that the limit is 50 watts.

4. The sample should not be heated to a temperature greater than 300 K (600 K if equipped with high temp. option).

5. Avoid contact with the cold gases.

6. Make sure that the storage dewar helium reservoir is equipped with a safety pressure relief. If needed, install a relief off the helium gas hose.

7. When not in operation, set the phase separator to its lowest position. Rotate the regular knob, located above the instrumentation housing, clockwise [looking down on the system from above] as far as the adjustment will allow. This allows for maximum flow conductance in the system during warm-up.
8. Never `break' transfer line vacuum with helium gas. Remember the WORKSTATION and transfer line vacuums are completely separate.

9. The insulating vacuum spaces are protected from over pressure by installed safety reliefs. The relief has been preset at the factory. NEVER SET TO A HIGHER PRESSURE.

10. If pumping on the liquid helium to operate below 4.2 K, do not use, if possible, the same vacuum pump for the insulating vacuum of the dewar. Large amounts of helium may temporarily contaminate the roughing pump causing helium to backstream into the insulating vacuum space of the dewar.

Wiring Information

WIRING

Unless otherwise noted, the following wiring scheme is used.
Temperature sensors are installed in a four wire configuration with
Pin A - Sensor I+
Pin B - Sensor V+
Pin C - Sensor V-
Pin D - Sensor I-

The Heater is attached to pins G-H.